A STUDY OF COMPLETE ANALYSIS OF RAW SALT USED FOR CAUSTIC SODA PRODUCTION

Work done at
TRAVANCORE COCHIN CHEMICALS (TCC) LTD

Project work submitted to
MAHATMA GANDHI UNIVERSITY ,KOTTAYAM
In partial fulfillment of the requirement for the
award of the degree of Bachelor of
Science in Chemistry

By
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DEPARTMENT OF CHEMISTRY
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MURICKASSERY
2020-2023

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CERTIFICATE

This is to certify that the project work entitled "**A STUDY OF COMPLETE ANALYSIS OF RAW SALT USED FOR CAUSTIC SODA PRODUCTION**" is an authentic work carried out by Mrs. GOURI SURESH (Reg.No: 200021026731),in partial fulfillment of the requirements for the award of the Degree of Bachelor of Science in Chemistry in Mahatma Gandhi University, under my supervision and guidance during the academic year 2020-2023

Prof. SAJI K JOSE
Head of the Department
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Place: Murickassery
Date:

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CERTIFICATE OF PROJECT WORK

This is to certify that ***Ms. Gouri Suresh (Reg. No. 200021026722)****, a student of B.sc Chemistry from Department of Chemistry, Pavanatma College, Murickassery - 685604, has successfully completed Project Work in* ***"A Study of complete analysis of raw salt used for Caustic Soda production"*** *in our company from 23.05.2022 to 01.06.2022.*

We wish all the best for her future.

Shibu S. Dev
Manager (HR)

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DECLARATION

I, GOURI SURESH, hereby declare that the dissertation entitled **"A STUDY OF COMPLETE ANALYSIS OF RAW SALT USED FOR CAUSTIC SODA PRODUCTION" SUBMITTED TO THE UNIVERSITY OF KOTTAYAM** in partial fulfillment of the requirement for the award of Degree of Bachelor of science in chemistry is a record of original project done by me during the period 2020-2023. This report has not been submitted earlier to this University for the fulfillment of the requirement of a course of study.

GOURI SURESH

Place: Udyogamandal
Date :

ACKNOWLEDGEMENT

The satisfaction that accompanies any task would be incomplete without mentioning the people, whose constant guidance and encouragement have served as a beam of light and crowned my efforts with success.

I would like to express my sincere thanks to the Principal, **Pavanatma College, Murickassery** who has given me an opportunity to carry out my thesis work in one of the most prestigious institutes.

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Above all I thank the God Almighty for giving unlimited support and encouragement for the successful completion of this project work.

Gouri Suresh

CONTENTS

CHAPTER 1

INTRODUCTION

Caustic soda is an alkali salt which is also called Lye. It is the common name of sodium hydroxide . This name is given due to the corrosive nature of this salt on animal and plants tissues. It has a wide range of application. Caustic soda is an important chemical in the manufacture of clothes, paper, soap etc which are essential commodities of our day to day life. It is also used for the manufacture of Rayon, Nylon, Caprolactam etc.The main raw material used for the production of caustic soda is common salt (NaCl). Caustic soda is produced by the removal of impurities in the raw salt followed by the electrolysis.

INDUSTRIAL IMPORTANCE

Caustic soda is the basic material used in soaps and dye making. It is also a very useful compound for drugs, textiles, rubber, paper and petroleum industries. In ancient times, caustic soda was used for soap making only. Nowadays, it is a very useful industrial chemical for many industries. Large scale manufacture of caustic soda has been carried out after the advent of the electrolysis process in the latter half of the 19th century.

APPLICATIONS

Direct application (Pulp and paper, soap and detergents, alumina, petroleum, textiles, water treatment) organic chemicals (propylene oxide ,polycarbonate ethylene amines, epoxy resins), Sodium/Calcium hypochlorite, Sulphur contain compounds, Sodium cyanide etc. Sodium hydroxide is the principle strong base used in the chemical industry. It can be used also as a normalizing agent in petroleum refining.

GENERAL PROPERTIES

Pure Sodium hydroxide is a white solid available in pellets, flakes and granules. It is also available as 32% and 50% caustic soda solutions. It is very deliquescent and also readily absorbs carbon dioxide from the air. So it should be stored in an airtight container. It is highly soluble in water with liberation of heat. It also dissolves in ethanol and methanol. It is insoluble in ether and other non polar solvents. A Sodium hydroxide solution will leave a yellow stain on fabric and paper.

CHEMICAL PROPERTIES

Sodium hydroxide is an ionic compound, containing Sodium ion and hydroxide ion. The hydroxide ion makes Sodium hydroxide a strong base which reacts with acid to form water and corresponding salts. Example, with hydrochloric acid, NaCl and water are formed.

$$NaOH + HCl \rightarrow NaCl + H_2O$$

In general such reactions are represented by one simple net ionic equation.

$$OH + H \rightarrow H_2O$$

This type of reaction releases heat when a strong acid is used. Such acid base reactions can also be used for titration and indeed this is a common way for measuring concentration of acidic oxides such as SO_2. Sodium hydroxide slowly reacts with glass to form sodium silicates, so glass joints and stop cocks exposed to NaOH have a tendency to freeze, flasks and glass-fined chemical reactors are damaged by long exposure as glass becomes frozen. Sodium hydroxide does not attack Iron or Copper but many other metals such as Al, Zn and Ti are attacked rapidly.

In 1986 an Aluminium road tanker in the UK was mistakenly used to transport 25% NaOH solution causing precipitation of the contents and it damaged the tanker. For the same reason Aluminium pans should never be cleaned with lye.

$$2Al(s) + 6\,NaOH(aq) \rightarrow 3H_2(g) + 2Na_3AlO_3(aq)$$

Many non-metals also react with NaOH giving salt. For example Phosphorus forms Sodium phosphate. Unlike NaOH, the hydroxide of most metals is insoluble and therefore Sodium hydroxide can be used to precipitate metal hydroxide. One such hydroxide is Al(OH); and is prepared at the treatment plant from Aluminium sulphate by reaction with NaOH.

$$6NaOH\ (aq) + Al_2(SO_3)\ (aq) \rightarrow 2Al(OH)_3(aq) + 3Na_2SO_4(aq)$$

Sodium hydroxide reacts readily with carboxylic acid from their salts, and it is strong enough to form salts with phenols. NaOH can also be used for the base driven hydrolysis of esters (as saponification), acids and alkyl halides. However the limited solubility of NaOH in organic solvent means that the more soluble KOH is often preferred.

THE RAW MATERIALS USED FOR THE PRODUCTION OF CAUSTIC SODA

The main raw material used for the production of caustic soda is common salt (NaCl). About 3700 units of electricity is consumed for about 1.7 metric tons of caustic soda production.

THE IMPURITIES PRESENT IN SALTS

In natural occurrences salt is never found absolutely pure. Most of the deposits have been formed by deposition from sea miner amounts.water, which in addition to the salt contains many other dissolved and suspended substances as impurities. They are Calcium anhydride, Dolomite, Calcite, Pyrite, Quartz and Iron oxides. The most common soluble impurities include the following ions Ca^{2+} · Mg^{2+} · K+, Cr, Ba^{2+} ·Sr, Br may also be present in salt.

VARIOUS STEPS IN THE PROCESS OF MAKING CAUSTIC SODA

The caustic soda (NaOH) production mainly involves two stages.

1. The removal of impurities in the raw salt.
2. Electrolysis.

The raw salt used for electrolysis should be analyzed to estimate the amounts of impurities present in it. From this the amount of chemicals needed for the removal of impurities can also be calculated.

ESTIMATION OF IMPURITIES

The raw salt contains Ca^{2+}, Mg^{2+} and SO_4 as the main impurities. The Ca^{2+} and Mg^{2+} is estimated by complexometric titration with EDTA solution and SO_4 ions can be estimated as $BaSO_4$ by gravimetric estimation method. The insoluble residues and moisture in raw salt can be estimated for determining the composition of the salt.

REMOVAL OF IMPURITIES

1. CALCIUM

Calcium is present as Calcium sulphate in the raw salt. It is removed as Carbonate by treating the brine with soda ash (Na_2CO_3) in the dissolved form.

$$CaSO_4 + Na_2CO_3 \rightarrow CaCO_3 + Na_2SO_4$$

2. SULPHATE

Sodium sulphate in the brine is ordinarily due to the treatment of Calcium sulphate with Sodium carbonate. The common reactants to remove Sulphates are $BaCl_2$ and $BaCO_3$.

Now

$$SO_4 + BaCl_2 \rightarrow BaSO_4 + 2NaCl$$

$$Na_2SO_4 + BaCO_3 \rightarrow BaSO_4 + Na_2CO_3$$

Barium carbonate is preferred over $BaCl_2$ because of the lower reagent cost. The settled precipitate can be discarded. The Sulphate if present is more than 6 g/L will affect the membrane used for the electrolysis.

3. MAGNESIUM

Magnesium present as $MgCl_2$ and $MgSO_4$ in the brine solution can be precipitated by treating with caustic soda or hydrated lime. Caustic soda is used in TCC to avoid excess impurities problems with hydrated lime. Moreover if NaOH is used it produces NaCl as a byproduct and can be reused in the plant.

$$MgSO_4 + 2\ NaOH \rightarrow Mg(OH)_2 + Na_2SO_4$$

$$MgCl_2 + 2\ NaOH \rightarrow Mg(OH)_2 + NaCl$$

During the above treatment the Na_2SO_4 produced is removed by treating $BaCO_3$ as said earlier. The raw brine is treated with NaOH after adding $NaCO_3$. This is because the $Mg(OH)_2$ precipitate tends to settle rapidly. In doing so it traps the suspended, slow setting, finely divided crystalline precipitate of $CaCO_3$ and allows it to settle faster. Thus residual turbidity is decreased.

ELECTROLYSIS

There are three types of cell such as diaphragm cell, mercury cell and membrane cell used for electrolysis. Nowadays, mercury cells are not used because of the pollution problem. Compared to diaphragm cells, the membrane cells are more efficient and hence they are used in TCC for electrolysis.

REACTIONS AND ENERGY CHANGES

During electrolysis of sodium chloride, the following reactions occur.

At anode,

$$NaCl \rightarrow Na^+ + Cl^-$$

$$Cl \rightarrow \frac{1}{2} Cl_2 + e^-$$

At cathode,

$$H_2O + e^- \rightarrow \frac{1}{2} H_2 + OH$$

$$Na^+ + OH^- \rightarrow NaOH$$

Overall Reaction

$$NaCl + H_2O \rightarrow NaOH + \frac{1}{2} H_2 + \frac{1}{2} Cl,$$

The energy consumed in the electrolysis of brine is the product of current flowing and the potential of the cell. The minimum voltage required for the process may be derived from Gibbs Helmholtz equation, which expresses the relation between the electric energy and the heat of reaction of the system.

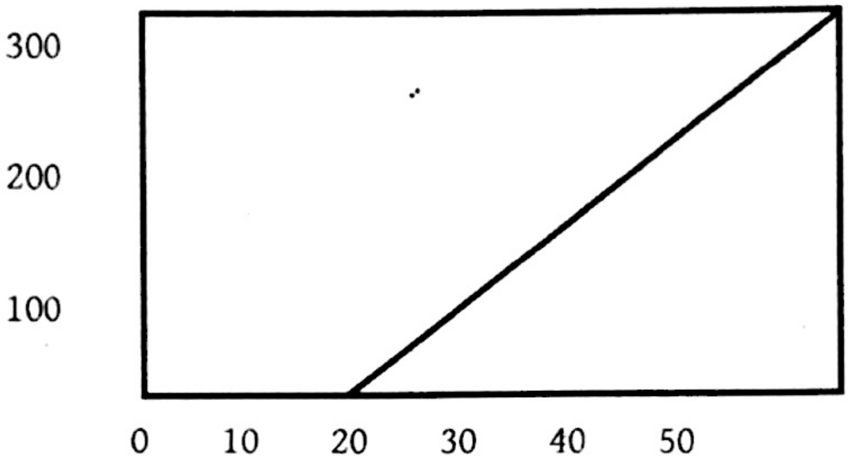
$$E = J\Delta H/F + TDE/dT$$

E -Theoretical decomposition voltage

ΔH - Enthalpy change of reaction

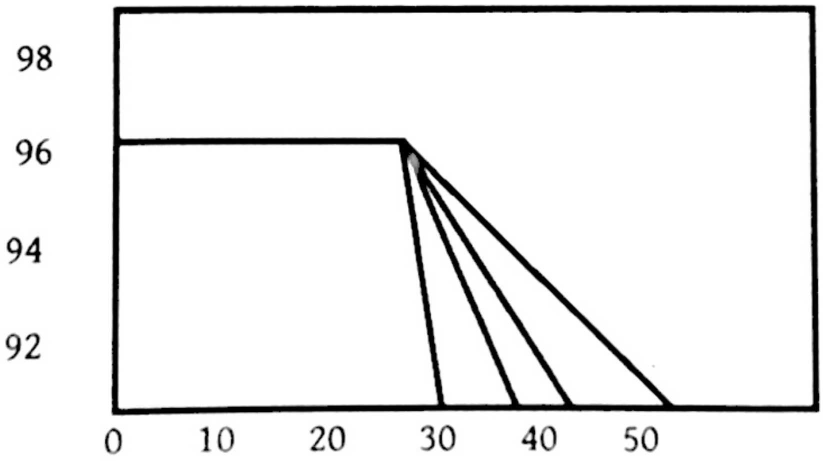
CALCIUM AND MAGNESIUM

Calcium and Magnesium are the most harmful substances to the membrane. The permissible level of Ca and Mg are independent of the concentration of the electrolyte. The relation between Ca^{2+} content in ppb and Ca^{2+} accumulation in membrane is given below;



X-axis - Ca^{+} Content in ppb
Y-axis - Ca^{2+} accumulation
(Kg/cm)

Relation between Ca content in ppb and current efficiency

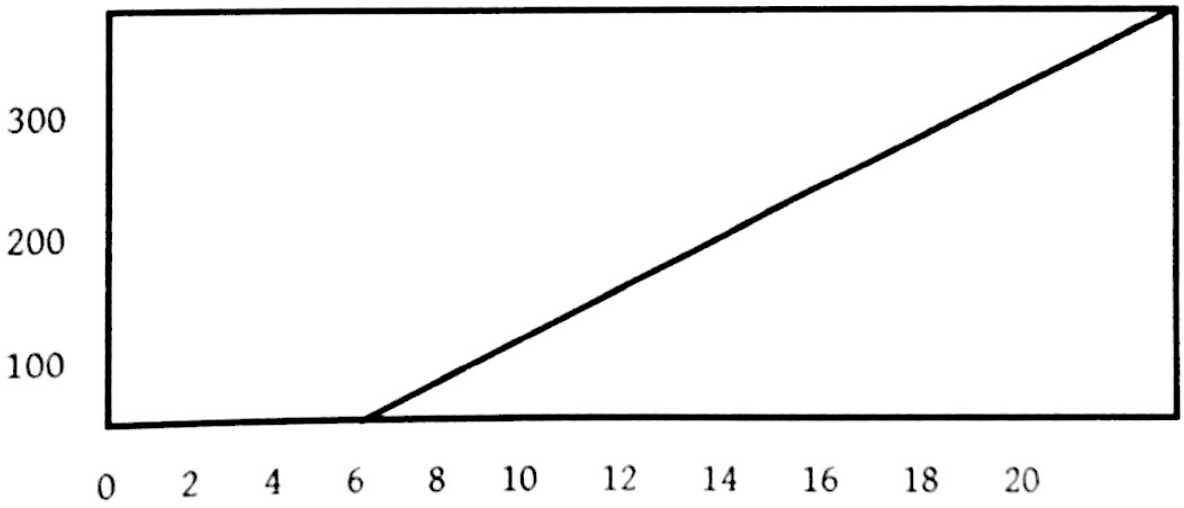


X-axis - Ca^{2+} content in ppb
Y-axis - Current efficiency

From the graph 1 and 2 it is observed that above 20 ppb of Calcium content in feed brine leads to the Calcium accumulation on the membrane and decreases the current efficiency of the membrane . Magnesium is lower in terms of harmfulness than calcium. The content of calcium combined with Magnesium should be 20ppb or less. The purity of high level feed brine is obtained by passing through the ion exchange resin.

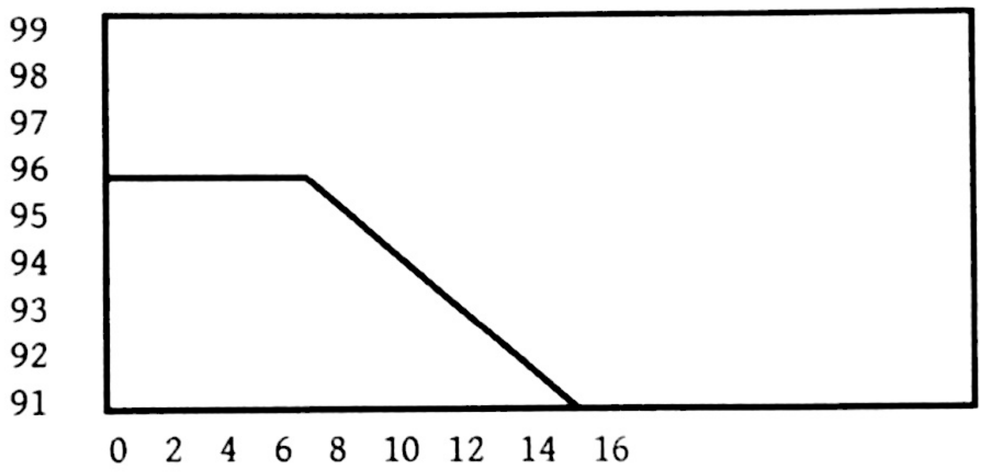
SULPHATE

If the Sulphate ion concentration in feed brine is higher than 6 g/L Sulphate ion is accumulated in the membrane and finally current efficiency will begin to go down. It is therefore important that the concentration of Sulphate ion in the feed brine is maintained at 6g/L or less



X-axis - SO_4^{2-} content in g/L
Y-axis - SO_4^{2-} accumulation
(Kg/cm)

Relation between SO_4^{2-} content in g/L
and current efficiency



MEMBRANE CELLS

Membrane cell consists of a semi permeable copolymer to separate the anode and cathode compartments. The membrane allows the passage of Sodium ions but rejects hydroxyl groups. Perfluoro sulphuric acid polymer (Nafion) is the first used membrane. The ASAHI glass company implemented bipolar cells with multiple layer membranes. It consists of perfluoro sulphuric acid polymer coated on one side with a fluoro carboxylic acid polymer. The membrane cell operates using a more concentrated brine and produces more pure and concentrated products (32% NaOH). Such products require only 7.15 Kg of water to be evaporated to produce metric tons of 50% caustic soda. The membrane cell consumes least electricity when compared with Mercury cell and Diaphragm cell and it also has 95% current efficiency. Membrane is the most critical component of the membrane cell technology and for current efficiency. Hence the cell voltage and hence energy consumption greatly depend on its quality

1. Ideal ion exchange membrane separators should have high selectivity of the transport of Na.
2. Negligible transport of Chloride, hypochlorite and chlorite ions.
3. Zero back migration of hydroxide ions.
4. Low electrical resistance.
5. Good mechanical properties and long term stability for practical use.

In TCC chlor alkali electrolyser consists of flemion type membrane and is a co-polymer of tetrafluoroethylene (Teflon) (CF_2 - CF_2) and perfluoro polymer(CF_2 - O-CF_2-CF_2-COO).

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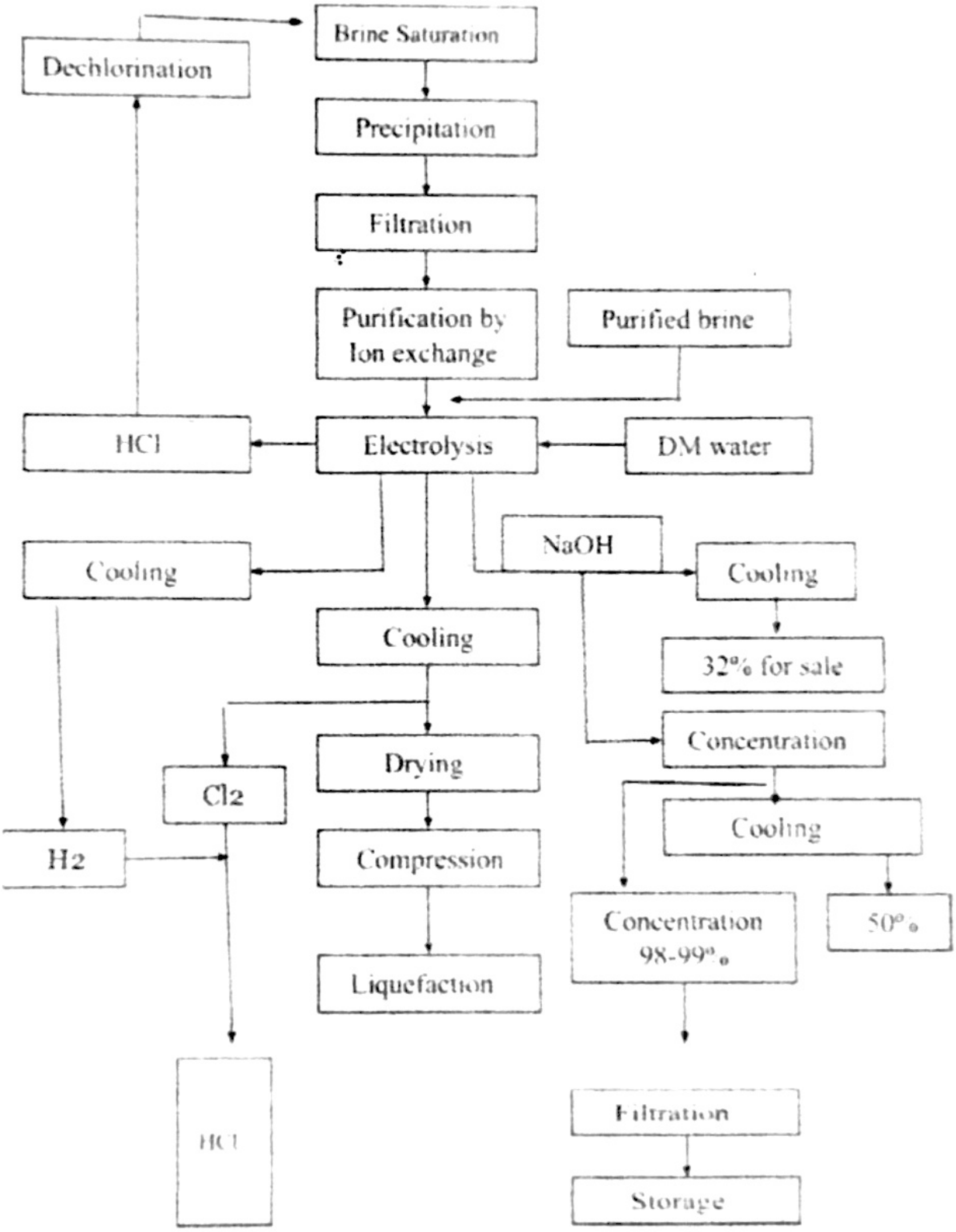
FLOW DIAGRAM OF THE MEMBRANE CHLOR ALKALI PROCESS



T - Absolute temperature

F - Faraday, constant coulombs/equivalent (96500)

N - Number of equivalent involved

The heat of reaction for the electrolysis of salt may be formed from the heat of formation of the components of the overall reaction or from the negative of these values, changes in the heat content of the system.

The overall reaction is;

$$NaCl(aq) + H_2O(l) \rightarrow NaOH(aq) + \tfrac{1}{2} H_2(g) + \tfrac{1}{2} Cl_2(g)$$

This may broken down to the following reaction for formation;

$Na(s) + \tfrac{1}{2} Cl_2(g) \rightarrow NaCl(aq)$	$\Delta H = -97.1$ Kcal
$H_2(g) + \tfrac{1}{2} O_2(g) \rightarrow H_2O(l)$	$\Delta H = -68.4$ Kcal
$Na(s) + \tfrac{1}{2} O_2 + \tfrac{1}{2} H_2(g) \rightarrow NaOH(aq)$	$\Delta H = -112.1$ Kcal

The net change in the enthalpy (ΔH) for the overall reaction is:

$$97.1 + 68.4 - 112.1 = 53.4\text{K Cal}$$

When this value of ΔH is substituted in the Gibbs Helmholtz equation and the change in voltage with temperature is neglected, the value of 'E' is found to be 2.31V. The omission TdE/dT involves an error of less than 10% for most cells. The efficiency of membrane cells is found to be 95%

IMPACT OF IMPURITIES ON PRODUCTION OF NaOH

During the ion exchange membrane electrolysis process, the feed brine must be pure, because in the presence of impurities, the membrane is more readily clogged and the current efficiency of the cell is reduced.

CHAPTER 2

AIM AND OBJECTIVE

In TCC, caustic soda is manufactured on a large scale. The main aim of the project work is to understand how the production of caustic soda is carried out and also to study the quality of the raw material. If the feed brine contains above 20 ppb levels of calcium and magnesium together and sulphate 6g / L leads to the clogging of the membranes and decreases the current efficiency of the membrane cell. So the impurities should be kept below that range. This can be achieved by the complete precipitation of impurities passing through the ion exchange resin. The excess dosage of chemicals makes the precipitation complete and removes all the impurities.

CHAPTER 3
EXPERIMENTAL METHODS

1.PREPARATION OF RAW SALT SAMPLE SOLUTION

About 50g of raw salt sample is weighed and taken in a 500mL beaker. Dissolve it in demineralised water, cooled and filtered the solution through counterpoised Whatmann No: 41 filter paper. The insolubles are washed till free from chloride. The filtrate is collected in a 500mL measuring flask and made up to the mark. The solution is used for all tests. The insoluble residue along with the filter paper and its blank is dried to a 110 degree celsius.

2. ESTIMATION OF INSOLUBLE RESIDUE

The filter paper with insoluble residue is heated in an oven for two hour at 110°C and weighed. The blank filter paper weight is also noted.

$$\text{Insoluble residue, \%} = \frac{\text{Weight of residue} \times 100}{\text{Weight of samp}}$$

3. ESTIMATION OF MOISTURE

Weigh out accurately about 1 to 1.5g of crushed salt into a porcelain dish which has been previously dried, cooled and weighed. Dry the dish with its content in the oven at 140°C for 2 hours, cool in a desiccator and find out the loss in weight. Do a duplicate analysis.

Moisture, % = Loss in weight x 100/weight of sample

After finding the loss in weight, the crucible with its contents is heated at 250°C in a hot plate for one hour, with the crucible closed with a lid. It is cooled in a desiccator and again the loss in weight is determined.

$$\text{Moisture, \%} = \frac{\text{Loss in weight} \times 100}{\text{Weight of the sample}}$$

4.ESTIMATION OF CALCIUM

EDTA forms a chelated soluble complex with certain metal cations. Certain dyes such as Patton and Reeders reagent are added to aqueous solution containing Ca^+ and Mg^{2+} at high pH range, the solution becomes vine red in color. If EDTA is added as a titrant, Ca^+ ion will be complexed. If sufficient EDTA has been added, a Calcium complex forms and the solution turns from vine red to blue in color.

At high pH range (12-14) the sample solution containing Mg^+ ions precipitate as $Mg(OH)_2$ so that only Ca^{2+} ions will react with Patton and Reeder's indicator complex. Hence from the titration with EDTA, we get the amount of Ca^{2+} ion present in the solution.

20 mL of made up sample solution is pipetted out into 250 mL conical flask. About 5 mL of 2N NaOH solution is added and diluted to 100mL by demineralised water. Add a pinch of Patton & Reeders indicator. The reaction mixture is titrated against the standard 0.01 M EDTA solution taken in the burette. The end point is the appearance of permanent blue color. The volume of EDTA consumed is noted as titre value (T.V).

$Ca^{2+}\% = T.V \times 40 \times 500 \times 100/100 \times 1000 \times 20 \times \text{wt of sample}$

5.ESTIMATION OF MAGNESIUM

When the solution containing Mg^+ and Ca^+ ions are titrated with EDTA in the presence of Eriochrome black-T, the EDTA first reacts with free Ca ions, then with free Magnesium ions and finally with Mg^+ indicator complex. Since the Mg^+ the indicator complex is wine red in color and the free indicator is blue at pH 7 and above, the color of the solution changes from wine red to blue at the end point. Mg^+ indicator complex + EDTA- Mg-EDTA complex + indicator +H

During this reaction, hydrogen ions are liberated, which lowers the pH of the solution and decreases the stability of Metal-EDTA complex. In order to overcome the pH change, a buffer solution is added.

20mL of sample solution is pipetted out into a 250 mL conical flask and diluted to 100 mL with hot demineralised water. SmL of buffer solution and 5 drops of Eriochrome black-T indicator are added. The reaction mixture is titrated against EDTA solution till the wine red color changes to clear blue with a greenish tinge. The volume of EDTA consumed is noted. From this subtract the EDTA volume consumed for calcium determination. The volume gives the T.V for Magnesium.

$$Mg^{2+}\ \% = \frac{T.V \times 24 \times 500 \times 100}{100 \times 1000 \times 20 \times \text{wt of sample}}$$

6.ESTIMATION OF CHLORIDES

When Chloride ions are titrated with $Hg(NO_3)_2$ solution in presence of Sodium nitroprusside indicator the $Hg(NO_3)_2$ solution is converted into Mercuric chloride.

The mixture is titrated against 0.1 N Hg $(NO_3)_2$ solution. End point is the appearance of slight turbidity. A duplicate analysis is conducted.

$$Cl,\% = \frac{T.V \times \text{Normality of } Hg(NO_3) \times 35.45 \times 500 \times 250 \times 100}{10 \times 1000 \times 20 \times 20 \times \text{wt of sample}}$$

7. ESTIMATION OF SULPHATE AS BARIUM SULPHATE

Sulphate ions in a definite volume of same solution are precipitated in presence of dil.HCI as $BaSO_4$, by adding $BaCl_2$, solution. In raw salt samples, sulphate is present as $CaSO_4$ $MgSO_4$.The precipitate formed is filtered off, washed with hot water, ignited and weighed as $BaSO_4$. The mass of Sulphate in the whole of the sample is calculated by knowing that 233.36g of $BaSO_4$ contains 96g of Sulphate.

About 100 mL sample solution is taken in a 250 mL beaker and about ImL 1:1 HCI is added and heated to boiling. About 20mL of 10% $BaCl_2$ solution is added dropwise with constant stirring. It is then boiled for about 5 minutes. The mixture is then cooled and supernatant liquid is tested for completion of precipitation by adding a few drops of $BaCl_2$ solution. If precipitation is completed, the solution is allowed to settle for 4hours.

The clear solution is filtered off through a Whatmann No.41 filter paper and the precipitate is washed with a small portion of hot water until the filtrate is free from chloride (test with $AgNO_3$). The precipitate is then transferred completely into the filter paper and washed again with hot water. After the water has drained off, the filter paper is removed from the funnel and folded in the form of a packet. It is placed in a previously weighed silica crucible, dried, the precipitate along with the filter paper is charred on a heater and the precipitate is incinerated at 850°C for half an hour in a muffle furnace. It is then cooled in a desiccator and weight of BaSO4. Conduct a duplicate analysis.

$$SO,\% = \frac{\text{wt of } BaSO_4 \times 96 \times 500 \times 100}{233.36 \times 100 \times \text{wt of sample}}$$

8. COMPUTATION OF NaCl

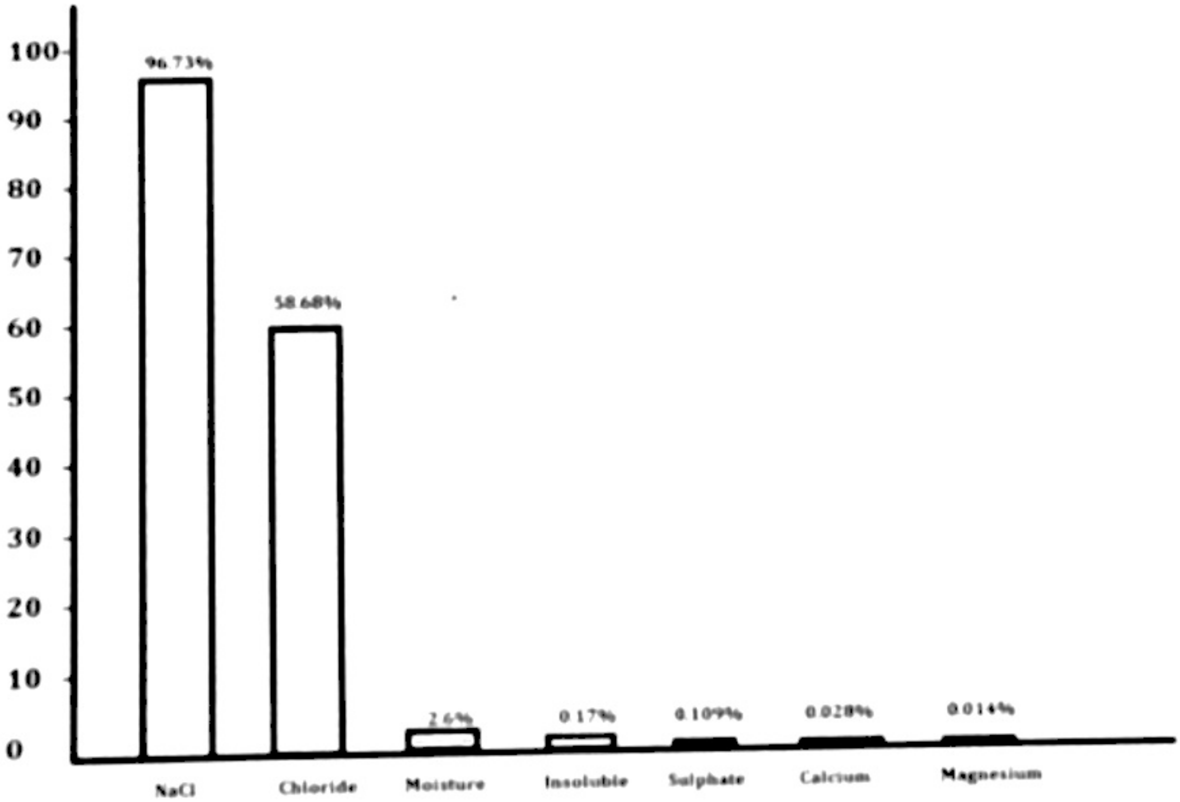
The raw salt contains Calcium as Calcium sulphate only. The Sulphates are present as Calcium sulphate and Magnesium sulphate and Chlorides as Magnesium chloride and Sodium chloride. This can be calculated as follows.

The total Calcium present as Calcium sulphate while Magnesium can be there in the form of Magnesium sulpahte and Magnesium chloride. To compute the Chloride due to Magnesium chloride first find out Sulphate due to Calcium. Deduct this Sulphate from total Sulphate. Find out Magnesium for the rest of Sulphate. Deduct this Magnesium from total Magnesium. The difference is the Magnesium which is present in the salt as Magnesium chloride. Find out Chloride due to this part of Magnesium. Deduct this Chloride from total Chloride. The remaining Chloride is known as residual Chloride and is due to Sodium chloride.

$$NaCl, \% = \frac{\%\ \text{residual chloride} \times 58.45}{35.45}$$

RESULTS AND DISCUSSION

Sl.No	PARAMETERS	SPECIFIED VALUE	OBSERVED VALUE
1.	NaCl	94% min	96.73%
2.	Chloride	---	58.68%
3.	Moisture	4% max	2.6%
4.	Insoluble Residue	0.4% max	0.17%
5.	Sulphate	0.48% max	0.109%
6.	Calcium	0.16% max	0.028%
7.	Magnesium	0.06% max	0.014%



From the above calculations, we get the probable composition of raw salt samples on a wet basis.

NaCl = 96.73%

Chloride = 58.68%

Moisture = 2.6%

Insoluble Residue = 0.17%

Sulphate = 0.109%

Calcium = 0.028%

Magnesium = 0.014%

The analyzed sample is acceptable because this contains allowed compositions of NaCl, impurity radicals, insoluble residues and moisture.

CONCLUSION

The experiments were carried out to find the amount of various impurities present in the raw salt that is used for caustic soda production. If the feed brine contains above 20 ppb level of Calcium and Magnesium and above 6 g/L of Sulphates lead to the clogging of membrane and decrease the current efficiency of the membrane cell. So the impurities should be kept below this range. This can be achieved by the complete precipitation of impurities and passing through the ion exchange resin. The excess dosage of chemicals than the theoretical makes the precipitation complete and removes all the impurities in time.

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